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Application Data Sheet

No.127A

GC-MS
Gas Chromatograph Mass Spectrometer

Simultaneous Analysis of 418 Pesticides Utilizing Smart SIM

The selected ion monitoring (SIM) method is capable of high-sensitivity measurements, so it is utilized for the analysis of trace components, such as residual pesticides in foods. With existing SIM conditions settings, however, the number of components is limited to a few hundred, and configuring the settings is difficult. In addition, when many ions are monitored simultaneously, the sensitivity is reduced. Smart SIM was developed to solve these problems. This article provides an example of its application to the simultaneous analysis of 418 pesticides.

Smart SIM

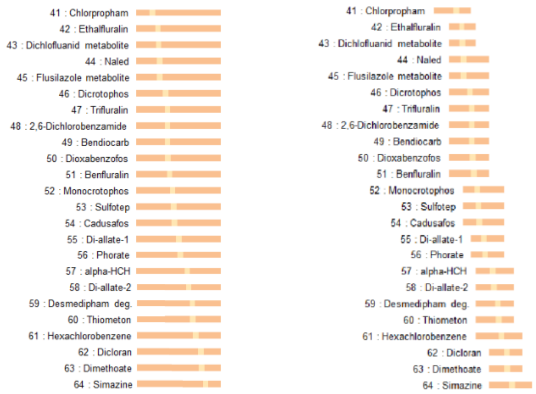


Fig. 1: SIM Measurement Time Program
(Left: Group Measurement Method;
Right: Measurement Method Using Smart SIM)
By using Smart SIM, the optimal time program synchronized to the retention times for each compound can be created.

As shown in Fig. 1, with the existing SIM method, a time program must be configured in order to collect data on designated ions only for designated time periods. If the number of target compounds exceeds 100, this time program becomes very complex. With the existing group measurement method (Fig. 1, left), configuring the settings is difficult, and sufficient sensitivity is not obtained because many ions are measured simultaneously.
In this example, a method was created utilizing Smart SIM, which automatically creates the optimal time program synchronized to the retention times for each compound (Fig. 1, right), and the sensitivity and accuracy were then evaluated.
In order to create a method with Smart SIM, the first step is to prepare the Smart Database (Fig. 2), which can be created simply by importing an existing method file if there is one. The retention index information is also registered; therefore, by utilizing the AART function with Smart Database, retention times can be estimated and adjusted without analyzing standard samples. Here, a method was created utilizing retention times adjusted with the AART function. The analytical conditions are indicated in Table 1.

(For details on retention time adjustments utilizing retention indices, check the Shimadzu website. http://www.an.shimadzu.co.jp/gcms/gcmssol/sol1.htm)

Serial#	Type	Acq. Mode	Compound Name (E)	Ret. Index 1	Ret. Time	Ion1			Ion2			Ion3		
						Type	m/z	Rati	Type	m/z	Rati	Type	m/z	Rati
41	Target	SIM	Chlorpropham	1666	10.228	T	213.0	100.00	Ref.1	127.0	219.30		154.0	56.14
42	Target	SIM	Ethalfluralin	1675	10.324	T	276.0	100.00	Ref.1	316.0	78.70		333.0	23.15
43	Target	SIM	Dichlofluanid metabolite	1676	10.334	T	200.0	100.00	Ref.1	92.0	128.69		108.0	22.13
44	Target	SIM	Naled	1678	10.355	T	185.0	100.00	Ref.1	109.0	595.24		145.0	152.38
45	Target	SIM	Flusilazole metabolite	1678	10.355	T	235.0	100.00	Ref.1	250.0	14.80		155.0	9.60
46	Target	SIM	Dicrotophos	1688	10.461	T	127.0	100.00	Ref.1	193.0	9.60		237.0	6.80
47	Target	SIM	Trifluralin	1691	10.493	T	306.0	100.00	Ref.1	264.0	88.68		335.0	8.18
48	Target	SIM	2,6-Dichlorobenzamide	1691	10.493	T	173.0	100.00	Ref.1	175.0	63.60		189.0	44.00
49	Target	SIM	Dioxabenzofos (Salithion)	1692	10.504	T	216.0	100.00	Ref.1	183.0	45.60		201.0	26.80
50	Target	SIM	Benfluralin	1695	10.535	T	292.0	100.00	Ref.1	264.0	24.40		293.0	12.40
51	Target	SIM	Monocrotophos	1700	10.588	T	127.0	100.00	Ref.1	97.0	18.00		192.0	10.00
52	Target	SIM	Sulfotep	1702	10.609	T	322.0	100.00	Ref.1	238.0	39.60		266.0	39.20
53	Target	SIM	Cadusafos	1703	10.619	T	159.0	100.00	Ref.1	158.0	69.20		270.0	5.20
54	Target	SIM	Di-allate-1	1711	10.702	T	234.0	100.00	Ref.1	236.0	39.73		86.0	254.79
55	Target	SIM	Phorate	1713	10.723	T	260.0	100.00	Ref.1	231.0	54.29		75.0	714.29

Fig. 2: Smart Database Screen

Table 1: Analytical Conditions

GC-MS:	GCMS-QP2020
Column:	SH-Rtx-5MS (30 m long, 0.25 mm I.D., df = 0.25 μm) (Shimadzu GLC, P/N: 221-75855-30)
Glass Insert:	Sky Single Taper Inlet Liner w/ Wool (Shimadzu GLC, P/N: 23336.5)

GC		MS	
Injection Port Temperature:	250 °C	Interface Temperature:	250 °C
Column Oven Temperature:	50 °C (1.0 min) → (25 °C /min) → 125 °C → (10 °C/min) → 300 °C (15 min)	Ion Source Temperature:	200 °C
Injection Mode:	Splitless	Ionization Mode:	EI
High-Voltage Injection:	250 kPa (1.5 min)	Measurement mode:	SIM
Injection Volume:	2 μL	Loop Time:	0.5 sec
Carrier Gas Control:	Linear velocity (47.2 cm/sec)		

Results

To create the sample, polyethylene glycol 300 was added as a pseudo matrix to a pesticides standard mixed solution to reach a concentration of 200 µg/mL. Fig. 3 shows the SIM chromatogram for the 100 ng/ml standard solution, and Fig. 4 shows the SIM chromatograms for each pesticide, as well as the %RSD and S/N ratios. Fig. 5 shows the results for the same sample, measured with a 244 component simultaneous analysis method via the existing group measurement method.

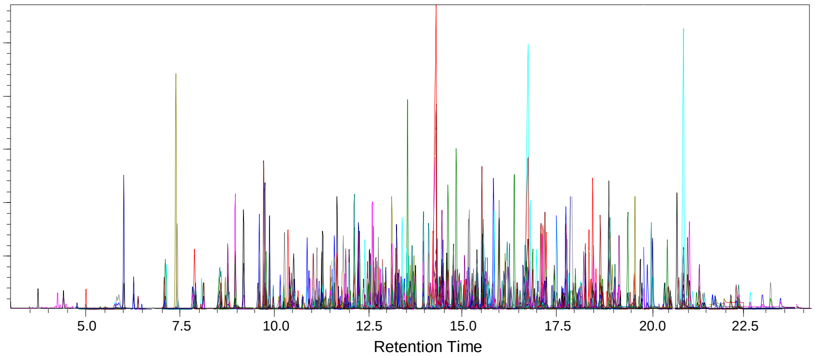
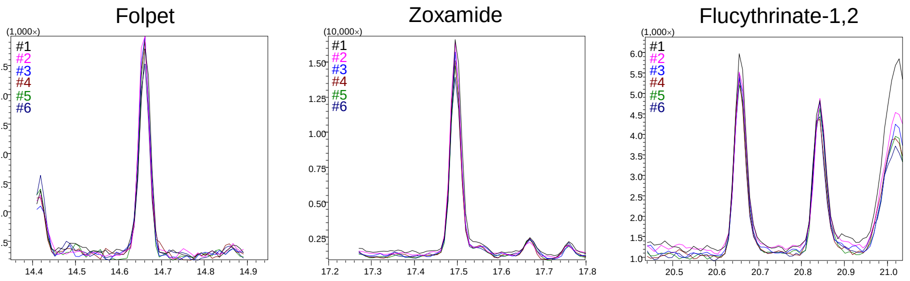
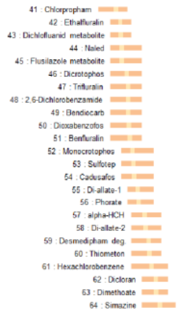


Fig. 3: SIM Chromatogram for a 100 ng/mL Pesticides Standard Mixed Solution



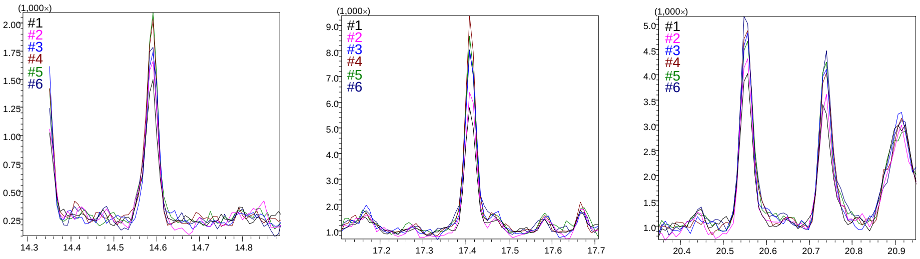


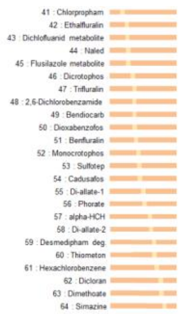
%RSD = 8.3 %
S/N = 35.6

%RSD = 5.9 %
S/N = 66.2

%RSD = 6.0, 4.8 %
S/N = 34.2, 25.8

Fig. 4: SIM Chromatograms, %RSD and Average S/N Ratios (n=6) for 2 ng/mL Sample





%RSD = 15.2 %
S/N = 16.0

%RSD = 24.0 %
S/N = 21.0

%RSD = 9.1, 11.3 %
S/N = 16.1, 11.3

Fig. 5: SIM Chromatograms, %RSD and Average S/N Ratios (n=6) for 2 ng/mL Sample with the Group Measurement Method

Using Smart SIM, a method was easily created that enables the simultaneous analysis of more than 400 components. In addition, it was evident that the analysis could be performed with higher sensitivity and higher accuracy in comparison to a method configured with the existing group measurement method. Further, as shown in Fig. 6, for more than 90 % of the 2 ng/mL components, the %RSD was 10 % max., indicating a highly accurate analysis.
Furthermore, with the existing method, to ensure sensitivity and accuracy, when the number of components exceeds 200, multiple measurement cycles are performed by dividing the method into several sections. In contract, Smart SIM allows obtaining the results with a single measurement. This can significantly reduce the analysis time. In addition, since the number of analyses per sample is reduced, so too are the frequency of maintenance and the cost, which will significantly improve laboratory productivity.

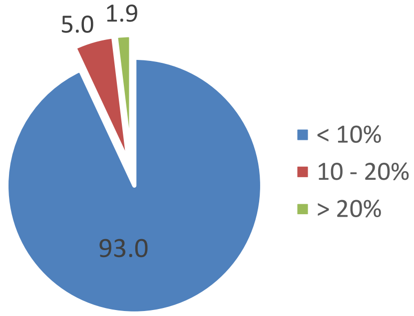


Fig. 6: %RSD Distribution for 2 ng/mL Sample
(Excluding oxpoconazole-formyl deg., which were not detected.)

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